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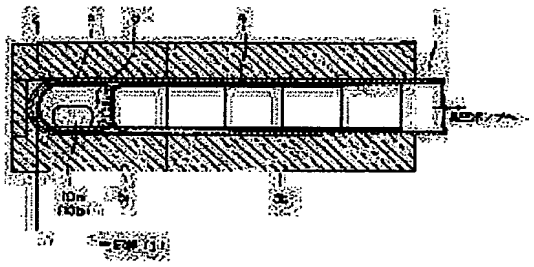
(54) APPARATUS FOR SEPARATING AND PURIFYING FULLERENE AND SEPARATION AND PURIFICATION

(57)Abstract:
PROBLEM TO BE SOLVED: To provide both an apparatus for separating and purifying fullerene capable of separating and purifying the high-purity fullerene at a low cost without using any organic solvent at all and a method for separating and purifying the fullerene.
SOLUTION: This apparatus for separating and purifying fullerene is equipped with at least a heating vessel 3 for heating the fullerene and subliming the fullerene, a trap 4, connected to the heating vessel 3 and used for depositing the sublimed fullerene and a vacuum device for decompressing and sucking the interiors of the heating vessel 3 and the trap 4. The heating vessel 3, trap 4 and vacuum device are arranged in the order. The trap 4 is preferably divided into two or more sections. The method for separating and purifying the fullerene comprises setting the temperature of the trap 4 on the side of the heating vessel 3 at a higher one than that on the side of the vacuum device.



CLAIMS

[Claim(s)]
[Claim 1] The separation refiner of the fullerene characterized by to arrange a heating container, a trap, and vacuum devices in this sequence while having at least the vacuum devices for making into a vacuum the interior of the trap for depositing the fullerene which connected with the heating container and this heating container for making the fullerene contained in fullerene content soot or rough fullerene heat and sublimate, and was sublimated, and said heating container and a trap.
[Claim 2] It is the separation refiner of the fullerene according to claim 1 which a heating container and a trap are held in the container of the shape of tubing which can stop an end and has opening in the other end, and is characterized by connecting said opening to vacuum devices.
[Claim 3] The separation refiner of the fullerene according to claim 1 or 2 which comes to allot a temperature control means to form in a trap the temperature gradient which turns the heating container side of a trap to an elevated temperature at the vacuum-devices side of Perilla frutescens (L.) Britton var. crispa (Thunb.) Decne., and serves as low temperature.
[Claim 4] The separation refiner of fullerene given in any 1 term of claims 1-3 characterized by for a trap dividing or more into two the fullerene sludge which deposited in this trap, and consisting of two or more disengageable partitions possible [ejection].
[Claim 5] The separation refiner of fullerene given in any 1 term of claims 1-4 characterized by a heating container and a trap consisting of heat-resistant ingredients, such as quartz glass, ceramics, and stainless steel.
[Claim 6] It is the separation refiner of fullerene to any 1 term of claims 1-5 characterized by coming to match the connection of a heating container and a trap the packed bed of a fiber.
[Claim 7] The separation purification method of the fullerene characterized by leading to the trap to which the temperature gradient which carries out temperature lowering of the fullerene which was made to heat and sublimate the fullerene contained in fullerene content soot or rough fullerene under a vacuum ambient atmosphere, and was this sublimated along the migration direction was given, and depositing it.
[Claim 8] The separation purification method of the fullerene according to claim 7 characterized by being the degree of vacuum whose vacuum ambient atmosphere is 1torr - 1x10 to 3torr.
[Claim 9] The temperature gradient of a trap is the separation purification method of the fullerene according to claim 7 or 8 characterized by being given so that the end side may be set to 500-650-degreeC and the other end side may be set to 200-300-degreeC.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the separation refiner and separation purification method of fullerene.
[0002]
[Description of the Prior Art] Fullerene is the allotrope of the carbon which was just discovered and had spherical-shell-like structure recently as 3rd carbon solid-state molecule which ranks second to graph AITO and a diamond. The structure of the fullerene C60 representing fullerene of a carbon number 60 is 32 face pieces the 12th page and whose forward hexagons a regular pentagon is the 20th page like a football. As fullerene, C60, the fullerene C70 of a carbon number 70, and the fullerene C84 of a carbon number 84 mainly exist. Although things for which all have cage-like structure with a unique form, such as C76, C78, C82, C90, and C94, are known as with a carbon number of 100 or less fullerene in addition to this, there are extremely few the amounts compared with C60. Moreover, existence of the fullerene of the macromolecule exceeding a carbon number 120 is also checked.
[0003] Such fullerene is useful at the following points.
(1) Utilization as a derivative by the chemical modification of fullerene : the attempt for which reactant high electrons are surrounding the outside of a globular form molecule, and C60 invents other matter and the matter which is easy to react chemically, and which carried out thing utilization, compounded various derivatives, and had a new function is made. For example, functional new plastics is reclaimed by introducing fullerene into a part of polymer, or it is in the phase used as a catalyst, and has succeeded in hydrogenating an acetylene series compound efficiently to a propylene system compound paying attention to the property in which metaled complex carries out occlusion of the hydrogen to C60. Moreover, C60 and C70 are packed into the hole of the porous material called a zeolite, and alcohols etc. can be compounded efficiently. Furthermore, as for the palladium catalyst which made fullerene support, several times of activated carbon are obtained, as for the hydrogenation effectiveness of a heptane.
(2) Application to a remedy : in order to reform that C60 melts only into organic solvents, such as benzene, water solubility is given by making the derivative which made the substituent which gets used to water, such as alcohol, add to fullerene. In these derivatives, the property to cut the specific part of DNA which is the body of a gene by the exposure of the property and light which check the activity of a proteolytic enzyme indispensable to the manifestation of HIV which caused the acquired immunode-ficiency syndrome is discovered.
[0004] (3) Utilization as a nonlinear optics functional material : the electronic transition complex which the compound containing fullerene and an electron donor forms discovers the Miyoshi nonlinear optical effect.
(4) Utilization which is ** as metallofullerene : although the crystal of C60 shows semiconductance, when alkali metal etc. is added, it becomes a metal or superconduction is shown. Moreover, it will also become a ferromagnetic if the organic amine system matter is

mixed.
(5) Other utilization : it is known that the optoelectric transducer which carried out the laminating of the special organic thin film on the thin film of C70 can be made as an experiment, the sensibility which exceeds a commercial item when the electrophotography photoconductor drum of the size which can be carried at a commercial small copying machine is produced can be attained, and an image sample can be obtained at the rate of per minute 20 sheets in A4 paper. Moreover, if C60 is added to the cell of the end differentiation before becoming hand and foot according to the trial within a test tube for the effect C60 affects the cell of a rat or a mouse, it is expected that chondrocyte increases by 4 usual times, a cartilage will grow if the population bone which was able to do C60 in the plastics metallurgy group is coated, and biocompatibility will go up. Furthermore, it is used also as ordinary temperature and a diamond composition ingredient under high voltage. Thus, since it is related to various application fields, fullerene is the matter useful as new materials, a remedy, etc.
[0005] Next, the conventional manufacturing method of fullerene is described. By heating by laser, the manufacturing method of fullerene evaporated graphite, was compounded by introducing inert gas, such as helium and Ar, into this ambient atmosphere, and accepts existence of fullerene at the time of discovery. However, this approach is not suitable in order to generate the soot of a large quantity, and as for current [many], arc discharge is used. By making a carbon rod (weight of 4.6g) with a diameter [of 5-6mm], and a die length of 1000-200mm into an anode plate, using a carbon rod also for cathode using 50-100A, and the DC power supply of 20-40V (20kW), and causing arc discharge in helium ambient atmosphere (100-400torr), the carbon rod of an anode plate evaporates and soot generates an example of the manufacturing method. Generally fullerene is contained in this soot five to 10% of the weight. And what separated fullerene from this soot is called rough fullerene.
[0006] Moreover, the approach of using the approach or plasma discharge which compounds fullerene from the soot which carried out high-frequency heating of the graphite rod, and carried out evaporation generation at 2700 degrees C, and compounding fullerene in helium ambient atmosphere, is tried by JP,5-116923,A etc. 40% according [the highest generation yield] to plasma discharge is reported on reference among these.
[0007]
[Problem(s) to be Solved by the Invention] The cutback of the manufacturing cost of fullerene is an important element which heightens the competitive strength of a product made from fullerene, and although it is becoming a low price every year, fullerene has the problem that it is still high as much as noble metals.
[0008] It has not succeeded in there being many unknown points also in current about the generation device of fullerene, and making C60 and C70 grade generate selectively. Therefore, it is desirable to carry out separation purification of the fullerene of C60 and C70 grade at high concentration, respectively from rough fullerene, such as the above-mentioned soot containing various kinds of fullerene of C60 and C70 grade. Since the clear application is not fully established since the large quantity manufacturing method of fullerene is discovered and there is also no between or, it is not clear which fullerene is needed by the aforementioned field of the invention among fullerene with many classes. However, since a strange property continues to be expected from fullerene, it is desirable to carry out separation purification of the specific fullerene of C60 and C70 grade from rough fullerene, respectively.
[0009] Although energy has been directed towards check identification of the fullerene by the mass spectrometer (MS) by the researcher of the beginning many, research of the fullerene related field is almost impossible only at the check by the mass spectrometer. It becomes indispensable that the fullerene of carbon number regularity comes to hand from the fullerene which has various structures in measurement of bioactive at a certain amount stability to research pans, such as physical-properties research represented by superconduction, research of the catalysis in a chemical reaction, and chemical property. In

order to realize this, the approach of carrying out separation purification of the specific fullerene from carbonaceous soot etc. is required.
[0010] As this separation purification method, the separation technology called liquid chromatography (LC) is known. This separation technology is the approach by which fullerene used dissolving it although solubility is not so large for several sorts of organic solvents (benzene, toluene, a hexane, carbon disulfide, etc.). There is a method of feeding a moving-bed liquid with the high performance pump called HPLC (high performance pump liquid chromatograph), and attaining high-resolution separation from the separation method which used the natural drop called a column chromatography for liquid chromatography, etc. In the aliquot by the column chromatography, if the amount of preparative isolation increases, since the amount of solvents to be used will become abundant, preparation of an organic solvent, recovery, and management take much time amount, and it becomes a factor with the big cost high. since [moreover,] separability is low -- the 74 or more C various limousine taxi fullerene C74 -- < -- it is not effective in separation purification.
[0011] If selection with the column of silica gel and mobile phase which, on the other hand, carried out various chemical modification including the octadecyl silane (ODS) column which is a chemical bond mold silica by the separation purification by the HPLC method is performed, preparative isolation of fullerene and separation purification will be possible, and it will be a separation means indispensable to separation of limousine taxi fullerene. However, it is not easy to carry out separation purification of the non-refined fullerene, such as a lot of rough fullerene, efficiently at once, and it has the problem that the chemistry actuation covering a multistage story is required until it collects the fullerene of a constant rate.
[0012] On the other hand, the approach of carrying out separation purification only of C60 selectively by the flash plate chromatograph using activated carbon was developed in the U.S. in 1992. According to this approach, the amount of a solvent is 1/20 or less [conventional], and has attained the laborsaving also with separation purification time amount larger than 1/5 to 1/30 or less, and the separation purification method by the HPLC method. If aimed only at separation of C60, it can be said to be one of the best approaches, but since the adsorption power to the activated carbon of fullerene is very large, separation of those other than C60 has an inapplicable fault.
[0013] Although separation purification of the fullerene by liquid chromatography is put in practical use as a large quantity approach, the problem is that it is not desirable on an environment and economy to use a moving-bed solvent for a large quantity. Moreover, in order to influence of a solvent the fullerene which that fullerene is very activity refined by the deposit from a solvent in growth of a single crystal by becoming clear, it has the problem that pure fullerene is hard to be obtained.
[0014] As mentioned above, as explained, development of the fullerene separation purification method which does not use an organic solvent is dramatically meaningful. As one of the fullerene separation purification methods, it is applied by ABERITTO and others (Averitt). FIJIKUSU Although the report has accomplished on the letter (Appl.Phys.Lett., Vol.65, No.3, 18 July 1994) etc., it is the separation approach by the molecular distillation using the principle of KUNUZEN, and equipment is complicated and this approach has it to a lot of efficient processing. [unsuitable]
[0015] Let it be a technical problem for this invention to offer the separation refiner and separation purification method of fullerene by which danger or toxicity is accompanied and which do not use a lot of organic solvents at all while being made in view of said situation and carrying out separation purification of the fullerene of a high grade cheaply extremely.
[0016]
[Means for Solving the Problem] A heating container for invention according to claim 1 to make the fullerene contained in fullerene content soot or rough fullerene heat and sublimate, While having at least the vacuum devices for making into a vacuum the interior of the trap for depositing the fullerene which connected with this heating container and was sublimated, and said heating container and a trap It is the separation refiner of the fullerene

characterized by arranging a heating container, a trap, and vacuum devices in this sequence.
[0017] A heating container and a trap are held in the container of the shape of tubing as for which invention according to claim 2 can stop an end and which has opening in the other end, and said opening is the separation refiner of the fullerene according to claim 1 characterized by connecting with vacuum devices.
[0018] Invention according to claim 3 is the separation refiner of the fullerene according to claim 1 or 2 which comes to allot a temperature control means to form in a trap the temperature gradient which makes the heating container side of a trap an elevated temperature, and serves as low temperature towards the vacuum-devices side.
[0019] Invention according to claim 4 is the separation refiner of fullerene given in any 1 term of claims 1-3 characterized by for a trap dividing or more into two the fullerene sludge which deposited in this trap, and consisting of two or more disengageable partitions possible [ejection].
[0020] Invention according to claim 5 is the separation refiner of fullerene given in any 1 term of claims 1-4 characterized by a heating container and a trap consisting of heat-resistant ingredients, such as quartz glass, ceramics, and stainless steel.
[0021] Invention according to claim 6 is the separation refiner of fullerene at any 1 term of claims 1-5 characterized by coming to match the connection of a heating container and a trap the packed bed of a fiber.
[0022] Invention according to claim 7 is the separation purification method of the fullerene characterized by leading to the trap to which the temperature gradient which carries out temperature lowering of the fullerene which was made to heat and sublimate the fullerene contained in fullerene content soot or rough fullerene under a vacuum ambient atmosphere, and was this sublimated along the migration direction was given, and depositing it.
[0023] Invention according to claim 8 is the separation purification method of the fullerene according to claim 7 characterized by being the degree of vacuum whose vacuum ambient atmosphere is 1torr - 1x10 to 3torr.
[0024] Invention according to claim 9 is the separation purification method of the fullerene according to claim 7 or 8 characterized by giving the temperature gradient of a trap so that the end side may be set to 500-650-degreeC and the other end side may be set to 200-300-degreeC.
[0025]
[Embodiment of the Invention] Drawing 1 is the sectional view of the example of 1 operation gestalt of the separation refiner of the fullerene of this invention. The heating container 3 for the separation refiner of the fullerene of this invention heating the fullerene contained in fullerene content soot 10a or rough fullerene 10b, and making it sublimate, While having at least the vacuum devices for making into a vacuum the interior of the trap 4 for depositing the fullerene which connected with this heating container 3 and was sublimated, and said heating container 3 and a trap 4, the heating container 3, a trap 4, and vacuum devices are arranged by the serial in this sequence. And in the example shown in drawing 1 , the heating container 3 and TORAPPUPU 4 are held in the container 2 of the shape of tubing which can stop an end and has opening in the other end.
[0026] A container 2 is a pressure-resistant container for holding the heating container 3 and TORAPPUPU 4 in the interior, making the interior of the heating container 3 and TORAPPUPU 4 into a vacuum, and heating these. A container 2 can stop the end and opening of the other end is connected to a vacuum-devices side. Since it is not necessary to connect so that vacuum leakage may not produce the heating container 3 and a trap 4 if the heating container 3 and a trap 4 are held in the interior of a container 2, it is easy to make these interior into a vacuum. Moreover, a separation refiner can be miniaturized if the heating container 3 and a trap 4 are put in order and held in a container 2. The desirable configuration of a container 2 is the tubular object by which the end was stopped, and an example of the magnitude is 5-25mm in tube lengths 0.5-1m and outer diameter. As for a container 2, what bears the temperature of 1000-degreeC is desirable, and it is desirable to

be constituted with heat-resistant ingredients, such as a quartz, ceramics, and stainless steel, especially.

[0027] The heating container 3 is a container for making the fullerene contained in fullerene content soot 10a or rough fullerene 10b heat and sublimate while holding charges of fullerene purification lumber, such as fullerene content soot 10a or rough fullerene 10b, in the interior. And the heating container 3 can stop the end and has opening for connecting with a trap 4 in the other end. The heating container 3 is heated from the method of outside through container 2 grade by heating means, such as electric furnace 5a. As for the heating container 3, it is desirable to be constituted with heat-resistant raw materials, such as a quartz, Pyrex, or stainless steel.

[0028] A trap 4 is a container for connecting with the heating container 3, making the fullerene sublimated and evaporated condense on the internal surface etc., and making it deposit. And as for a trap 4, it is desirable that it is a tubular object, and it has opening to ends.

[0029] A trap 4 is heated from the method of outside by heating means, such as electric furnace 5b, in order to make separation purification of fullerene efficient. Temperature control of the trap 4 is carried out so that a postscript may be carried out with a heating means to have a well-known temperature control means. Moreover, as for a trap 4, it is desirable that temperature control is carried out by the temperature control means which can form in a trap 4 the temperature gradient in which temperature falls toward the vacuum-devices side from the heating container 3 side of a trap 4. A trap 4 is constituted by heat-resistant raw materials, such as a quartz, Pyrex, or stainless steel.

[0030] Although a trap 4 can also be used as the heating container 3 and this body, it is desirable to use a trap 4 as the heating container 3 and another object. It is easy to take out another object, then a fullerene sludge from the inside of a trap 4. If it considers as another object, a trap 4 is divided further and it is made to constitute from two or more disengageable partitions, a fullerene sludge can be divided or more into two, and can be extracted. C70 [therefore,] -- < -- the product from which the content of the fullerene of C70 and C60 grade differs can be isolated preparatively easily.

[0031] Said trap 4 is preferably divided into 2-8 pieces. The trap 4 is constituted from five partitions which consist of five tubular objects divided into isometry by the example shown in drawing 1 . And all the partitions of a trap 4 have opening to ends. If the trap 4 is divided into two or more partitions in this way, two or more purification objects with which the content of the fullerene of C60 and C70 grade differs, respectively can be obtained by one separation purification actuation.

[0032] Vacuum devices are equipment for carrying out evacuation of the interior of a container 2, the heating container 3, and a trap 4, and making it a vacuum, and are vacuum pumps etc.

[0033] Furthermore, it is desirable to allot the connection of the heating container 3 and a trap 4 the packed bed 6 of a fiber. In case the packed bed 6 of a fiber makes the inside of the heating container 3 a vacuum ambient atmosphere after holding fullerene content soot 10a or rough fullerene 10b in the heating container 3, it is required in order to prevent that said soot which is impalpable powder, or said rough fullerene disperses. As a thing of a fiber, the cotton-like thing which bears the temperature of 1000-degreeC is desirable, and ceramic wool yarn, quartz glass wool, etc. which were excellent in thermal resistance especially are desirable.

[0034] If the heating container 3, a trap 4, and vacuum devices are arranged in this sequence, the fullerene evaporated within the heating container 3 which the container 2 stopped and was held in the side by being located will be flowed and moved only to an one direction toward the direction of vacuum devices from the inside of the heating container 3. And on the internal surface of the trap 4 by which temperature control was carried out etc., according to the vapor pressure of hula RAREN, various kinds of fullerene differs in a deposit location, and the trap (prehension) of the evaporated fullerene which flowed in the trap 4 is

carried out. that is, in order of the sequence of the carbon number of fullerene, for example, the fullerene exceeding a carbon number 70, (C70 — <), and C70 and C60, since fullerene differs in a location and deposits toward the vacuum-devices side (downstream of flow) of a trap 4 from the heating container 3 side (upstream of flow) of a trap 4, the purification object which contains specific fullerene in high concentration can be obtained.

[0035] The separation purification method of the fullerene of this invention is the separation purification approach of leading to the trap 4 to which the temperature gradient which carries out temperature lowering of the fullerene which was made heated and sublimating the fullerene contained in fullerene content soot 10a or rough fullerene 10b under a vacuum ambient atmosphere, and was this sublimated along the migration direction was given, and depositing the this sublimated fullerene in a trap 4.

[0036] The separation purification method of fullerene in the case of using the separation refiner shown in drawing 1 is described in more detail below. Fullerene will be sublimated if heated under a vacuum. Therefore, fullerene tends to sublimate the interior of the heating container 3 and a trap 4 with vacuum devices as a degree of vacuum of 1torr - 10 to 3torr (Torr).

[0037] Furthermore, the fullerene contained in fullerene content soot 10a or rough fullerene 10b will be sublimated if it is heated by electric furnace 5a etc. through a container 2 and heating container 3 grade at 400-1000-degreeC. If a sublimation rate is slow in it being under 400-degreeC and 1000-degreeC is exceeded, the sublimation rate of fullerene will become excessive and it will become easy to mix rough fullerene in a sludge. As for the heating container 3, it is desirable it is not only to heat with the constant temperature in said temperature requirement, but to heat to **** from near 400 degree C where sublimation of fullerene begins to take place to 1000 degrees C sublimated rapidly. Therefore, as for electric furnace 5b, it is desirable that program temperature control is carried out in this way. a in drawing 2 is a graph which shows an example of the temperature distribution of the heating container 3, and is the graph which showed the temperature in the distance l (it illustrates to drawing 1) and its location from the left end (the heating container 3 stopping section) of the heating container 3.

[0038] The fullerene evaporated within the heating container 3 passes the packed bed 6 of a fiber, and is introduced in a trap 4 from opening of the partition on the left-hand side of a trap 4. And it condenses and deposits under a vacuum ambient atmosphere on the internal surface of the trap 4 controlled by temperature lower than the heating container 3 etc. In order to raise whenever [separation purification / of fullerene], it is desirable that the temperature gradient which carries out temperature lowering along the migration direction of the sublimated fullerene can be given to a trap 4. That is, as for a trap 4, it is desirable that the temperature gradient which makes the heating container 3 side an elevated temperature more, and makes the vacuum-devices side low temperature more can be given, it is with an elevated-temperature and low temperature side, and it is desirable to establish the temperature gradient of 250-500-degreeC. If such a temperature gradient is established, it will be easy to deposit fullerene according to a carbon number in good order, and the fullerene of a high grade will be easy to be obtained.

[0039] Therefore, temperature by the side of the end of a trap 4 (heating container 3 side) is set to 500-650-degreeC, and a temperature gradient is prepared in a trap 4 by setting temperature by the side of the other end (vacuum pump side) to 200-300-degreeC. Thus, if the fullerene which evaporated the inside of TORAFFU 4 which established the temperature gradient is circulated, it will be easy to adjust the deposit location within the trap of the fullerene of C60 and C70 grade. Therefore, the purification effectiveness of fullerene is excellent. b in drawing 2 is a graph which shows an example of the temperature gradient of a trap 4, and is the graph which showed the temperature in the distance l and its location from the left end (the heating container 3 stopping section) of the heating container 3. In the example shown in b in drawing 2 , the temperature at the left end of a trap 4 (heating container 3 side) is about 505-degreeC, the temperature of the right end (vacuum pump side)

is about 200-degreeC, and the temperature gradient which carries out temperature lowering gradually toward a vacuum pump side from the heating container 3 side is given to the trap 4.

[0040]
[Example] Hereafter, this invention is explained in detail. In the following examples and examples of a comparison, all of an amount, %, and a ratio mean weight and weight % and a weight ratio. First, the relation between whenever [stoving temperature / of fullerene content soot 10a], and the amount of sublimation of fullerene was investigated as follows using the separation refiner shown in drawing 1 . Fullerene content soot 10a which contains fullerene 7.3% of the weight was placed into the heating container 3 of the shape of 3g and tubing, and the packed bed 6 was allotted by putting ceramic wool yarn in opening of the heating container 3 further. This heating container 3 was held in the back side of the pressure-resistant container 2 of the shape of tubing which stopped the end.
[0041] As an object for traps, five separated tubular objects which have opening were prepared for ends with isometry. The tubular object of these five individuals was stuck mutually, and it considered as the trap which has a disengageable partition from the four break sections. The trap 4 and the heating container 3 were connected through the packed bed 6 by inserting this trap into the tubing-like container 2, and making it stick with a packed bed 6.
[0042] Subsequently, evacuation of the interior of a container 2 was carried out with the vacuum pump, and the degree of vacuum of the ambient atmosphere of the heating container 3 and a trap 4 was made into 3x10 to 2torr. Under this vacuum ambient atmosphere, several point temperature was changed in the 500 to 900-degree C temperature requirement, and fullerene content soot 10a was heated by a unit of 3 hour at each temperature. The fullerene sublimated within the heating container 3 passed through the inside of a packed bed 6, was drawn in the trap 4, and condensed and deposited on the internal surface. 3 hours after, all the sludges that deposited the trap 4 in ejection and a trap 4 from the container 2 were collected, and the AUW was measured. In addition, the temperature of 550-degreeC and its right end (vacuum pump side) of the temperature at the left end of a trap 4 (heating container 3 side) was 300-degreeC. The relation between whenever [stoving temperature / of fullerene content soot 10a], and the fullerene AUW which deposited in the trap 4 is shown in drawing 3 .
[0043] When whenever [stoving temperature / of fullerene content soot 10a] was 500 degrees C so that drawing 3 may show, the amount of fullerene which deposited was 0.003g. With lifting of temperature, the amount of deposits increased and 0.22g fullerene deposited at 900 degrees C. This shows that about 100% of fullerene sublimated from 3g of said fullerene content soot, and it deposited in the trap 4. That is, in order to make the fullerene contained in fullerene content soot 10a sublimate efficiently, it turned out that what is necessary is just to heat fullerene content soot 10a to 500-900-degreeC.
[0044] The separation refiner shown in drawing 1 performed separation purification of fullerene, using - example 1-, next rough fullerene 10b 50mg. In addition, the weight ratio (C60/C70) of C60 and C70 of this rough fullerene 10b was 5. Rough 50mg fullerene 10b was placed into the heating container 3, and it heated at 650 degrees C by electric furnace 5a.
[0045] Temperature (however, temperature of the center section of the partition) of each partition of the trap 4 divided into six partitions was set to 550-degreeC, 510-degreeC, 450-degreeC, 415-degreeC, 380-degreeC, and 310-degreeC toward the right-hand side (vacuum pump side) partition from the left-hand side partition (partition by the side of the heating container 3). Separation purification of fullerene was performed like the above by exhausting the interior of a container 2 with a vacuum pump, drawing the fullerene which was made to sublimate the fullerene in the heating container 3, and was this sublimated in a trap 4, and depositing it under a vacuum ambient atmosphere. And after purification termination, the trap 4 was separated from the carriageway marking of ejection and a trap 4, the trap 4 was separated into six pieces from the inside of a container 2, and the fullerene sludge divided

into six was obtained by taking out a sludge from each partition. Drawing 4 is the graph which showed the relation between the temperature of the partition (zone) of a trap 4, and the weight ratio (C60/C70) of C60 and C70 in the sludge taken out from the partition.
[0046] C60/C70 of the sludge taken out from the partition of 550-degreeC were 3.5, C60/C70 of the sludge of a 510-degree C partition were 1.06, and C60/C70 of the sludge of the 310-degree C partition by the side of vacuum devices amounted to 275, and were 99.6 % of the weight as purity of C60. Namely, if it takes into consideration that C60/C70 of the rough fullerene before purification were 5, as for the sludge of a 310-degree C partition, it turns out that C60 is refined by the high grade. In addition, although remained in the heating container 3 after purification, C60/C70 are 2.7, and it also became clear to have changed a lot to the ratios 5-2.7 of C60/C70 of rough fullerene. Drawing 5 is drawing having shown the yield of the sludge of each partition. In addition, the yield of fullerene ** weight of the fullerene which deposited to each partition in the amount of 50mg of used rough fullerene sample 10b, and is the doubled value 100.
[0047] Using the separation refiner of this invention shown in - example 2- drawing 1 , 200mg fullerene content soot 10a was used, and separation purification of the fullerene was carried out on the following separation purification conditions. as said soot 10a, C70 used [the rate of C60] what is fullerene (C70 -- <) with the 3 remaining% higher order than C70 by 14% at 83%, including a little more than 7% of fullerene (a little more than 14mg).
[0048] In addition, make the degree of vacuum in the tubing-like container 2 into 0.03torr, and whenever [stoving temperature / of soot 10a] is considered as 640-degreeC regularity. The temperature (temperature of the center section of the partition) of each partition of the trap 4 formed in five partitions disengageable It was referred to as 505-degreeC, 440-degreeC, 395-degreeC, 360-degreeC, and 300-degreeC toward the right-hand side (vacuum pump side) partition from the partition on the left-hand side of a trap 4 (heating container 3 side), and separation purification time amount was made into 21 hours. Five kinds of sludges which differ in a deposit location were obtained from each partition which separated the ejection out of a container 2, and a trap 4 from carriageway marking, separated into five pieces, and separated the trap 4 by extracting a sludge after separation purification termination. the fullerene C60, C70, and C70 in each [these] sludge -- < -- the component analysis was performed using the high performance pump liquid chromatograph (HPLC), and the separation purification effectiveness was investigated.
[0049] drawing 6 -- C60, C70, and C70 in the temperature of a partition, and the sludge of each partition -- < -- it is the graph which showed content by percentage by weight. As shown in drawing 6 , the content of C60 was 83 - 95 % of the weight in the sludge of the partition of the temperature below 395-degreeC, and was 20% or less in the sludge of the partition more than 450-degreeC. On the other hand, it turns out that separation purification of it is dramatically carried out with 70% in the partition of 440-degreeC at the high grade although the concentration of C70 is 14 - 16% in the sludge of the partition of 300-degreeC, 395-degreeC, and 505-degreeC. That is, C60 was contained in the sludge extracted from the partition below 395-degreeC at the high grade, and C70 was contained no less than 70% in the sludge extracted from the partition of 440-degreeC.
[0050] C70 [moreover,] -- < -- like the sludge with which it did not accept into the sludge extracted from the partition of 300 so that drawing 6 might show existence, but existence was accepted in the sludge extracted from the partition of 360-degreeC to 505-degreeC, and temperature was extracted from the high partition -- C70 -- < -- content was high. In the sludge extracted from the partition of 505-degreeC, 70< 60% of C was also contained.
[0051] C60, C70, and C70 in the sludge extracted by the partition of a trap 4 from the partition as explained above -- < -- concentration (comparatively) -- large -- differing -- C70 -- < -- it turned out that separation purification of C70 and C60 is carried out according to the partition by the side of [an elevated-temperature side to] low temperature. C70 [namely,] -- < -- each fullerene of C70 and C60 was able to obtain what is extremely contained by the high grade by one separation purification actuation. The weight

of the sludge in each partition is shown in drawing 7 .
[0052] According to drawing 7 , for the amount of deposits of the partition of 1.5mg and 360-degreeC, the amount of deposits of the partition of 3.9mg and 395-degreeC was [the amount of fullerene deposits (the amount of traps) extracted from the partition of 300 degreeC / the amount of deposits of the partition of 1.0mg and 505 degreeC of the amount of deposits of the partition of 6.9mg and 440 degreeC] 0.9mg. The deposit AUW of the partition below 400-degreeC is 87% at the same time it occupies the moiety of 49% and the whole sludge near 400-degreeC, the amount of deposits of the partition more than 400-degreeC remains, and is 13%, and when it sees roughly, it turns out [of the fullerene C60 contained in rough fullerene] that they are 83% and the value comparatively near 14% of C70 comparatively.
[0053] moreover, the total amount of these sludges -- 14.2mg -- becoming -- rough fullerene 200mg -- the inner amount (a little more than 14mg) of fullerene sublimates about 100%, it deposits and it is shown that the trap was carried out. Even if compared with the fullerene content in the soot by solvent extraction being 7 % of the weight, sufficient rate of separation purification is shown.
[0054]
[Effect of the Invention] As explained above, it is the separation refiner and separation purification method which do not use a lot of organic solvents at all in order that the separation refiner and separation purification method of fullerene of this invention may perform all by the dry process, therefore while being able to reduce the separation purification cost of fullerene, it can work efficiently [it is safe and] safely. Furthermore, it is the separation refiner and separation purification method which can obtain the purification object which contains fullerene with a fixed carbon number in a high grade extremely in some numbers by one separation purification actuation. Moreover, since the trap is divided into two or more partitions, the sludge containing the fullerene from which a carbon number differs can be isolated preparatively easily.

[Translation done.]

* NOTICES *

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the sectional view of the separation refiner of the fullerene of the example of 1 operation gestalt of this invention.
[Drawing 2] It is the graph with which (a) shows an example of the temperature gradient of a heating container, and (b) shows an example of the temperature gradient of a trap.
[Drawing 3] It is the graph which shows the relation between whenever [stoving temperature / of rough fullerene], and the amount of sublimation of fullerene.
[Drawing 4] It is the graph which showed the weight ratio of C60 and C70 of the temperature of the partition of a trap, and the sludge of each partition.
[Drawing 5] It is the graph which showed the relation between the temperature of the partition of a trap, and the fullerene yield in each partition.
[Drawing 6] the fullerene C70 higher order than C60, C70, and C70 in the temperature of the partition of a trap, and the sludge of each partition -- < -- it is the graph which showed relation with a rate.
[Drawing 7] It is the graph which showed the temperature of the partition of a trap, and the weight of the sludge of each partition.
[Description of Notations]
2 [.. An electric furnace, 6 / .. The packed bed of a fiber 10a / .. Fullerene content soot, 10b / .. Rough fullerene] .. A container, 3 .. A heating container, 4 .. A trap, 5a, 5b

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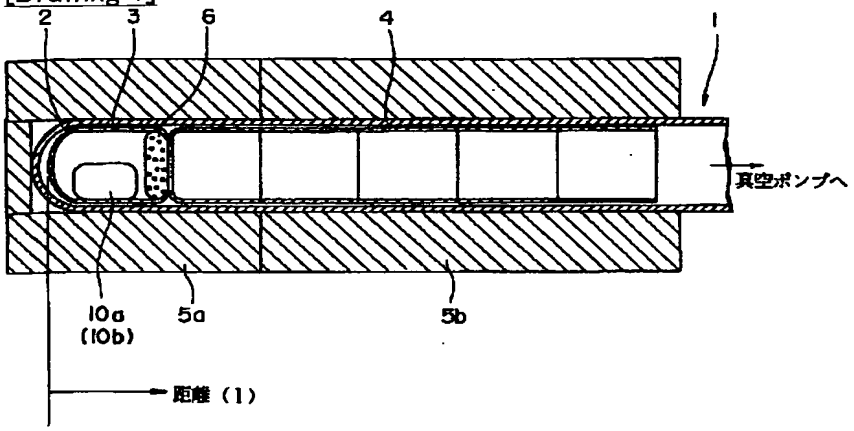
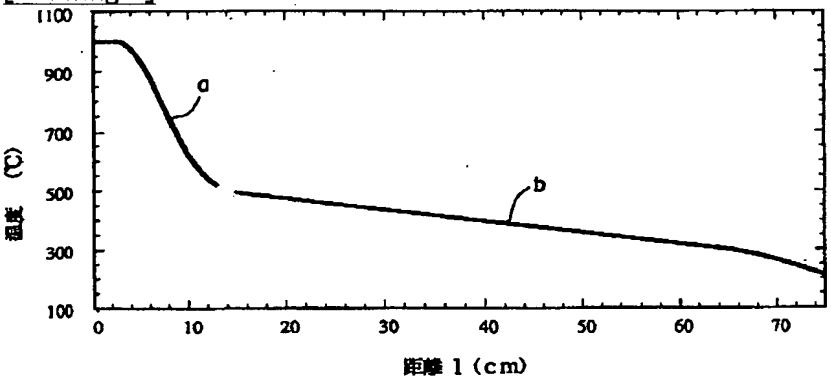
DRAWINGS

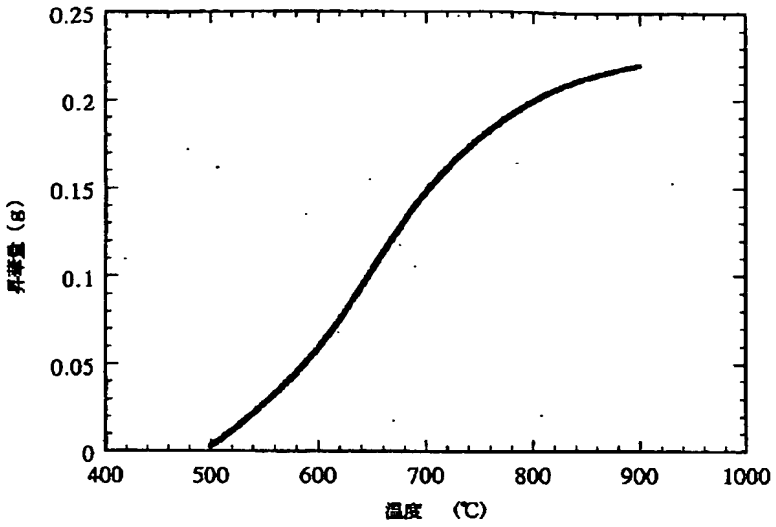
[Drawing 1]



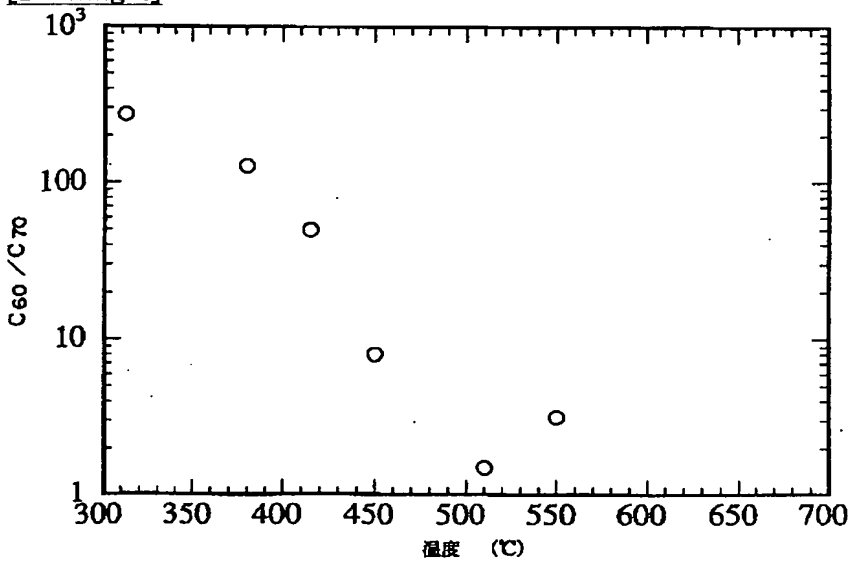
[Drawing 2]



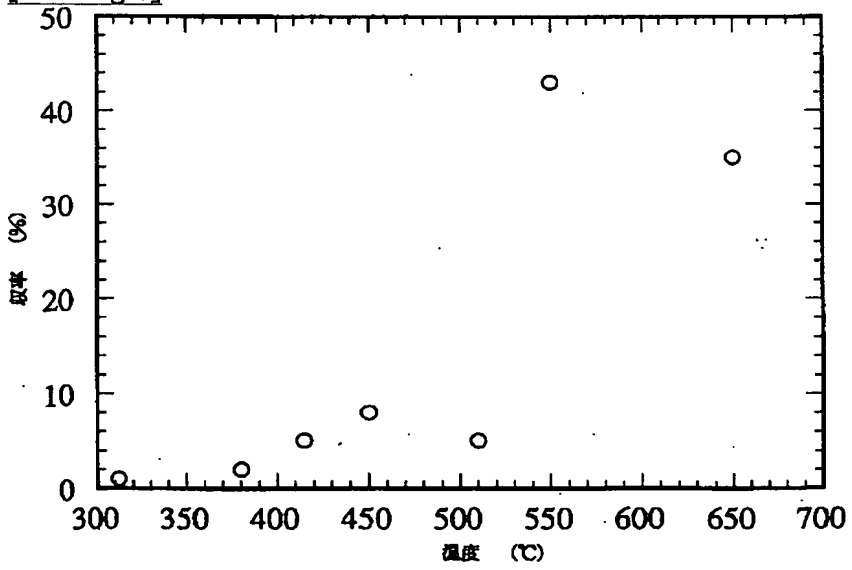
[Drawing 3]



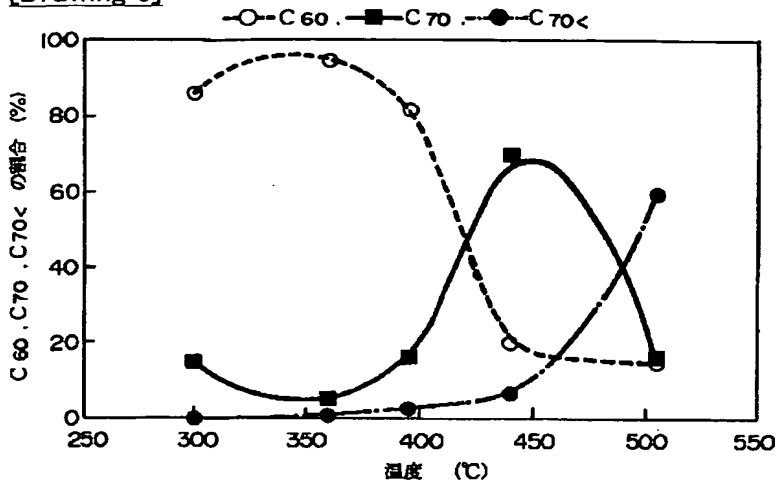
[Drawing 4]



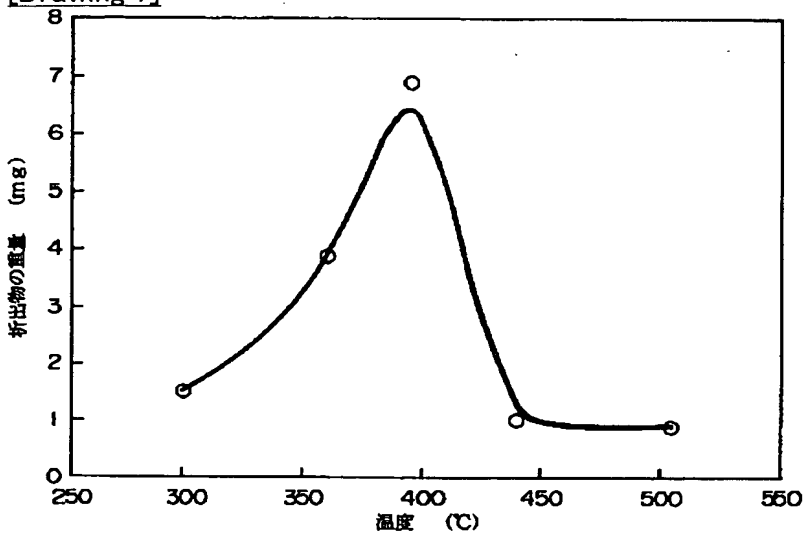
[Drawing 5]



[Drawing 6]



[Drawing 7]



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